

# LARGE ENVIRONMENTAL CHAMBER: AMMONIA RECOVERY CALIBRATION

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**ABSTRACT.** *The ability to accurately measure ammonia emissions from farms is an important issue both in terms of establishing emissions regulations and for effective evaluation of mitigation techniques. To address this issue, experimental trials were carried out to determine the ability to quantitatively recover ammonia released within a large environmental chamber designed to house six dairy cows or manure processing technologies. Ammonia was released over 30 min periods at a range of values (0.1 to 0.7 g/min) from three positions within the chamber. Chamber temperature was maintained at 22.2 °C. Air flow was maintained at one of three setpoints; 10.5, 14.0, or 21.0 air exchanges per h. The amount of ammonia recovered from each release was determined by calculating the average increase in ammonia concentration during the release and stabilization periods, and multiplying by the measured volumetric air flow. Over 86 trials, the recoveries averaged  $105.1 \pm 0.8\%$  of the amounts released as determined by the weight change of the release cylinder. Small increases in measured recoveries were associated with increased air flow and increased amounts of ammonia released. Results from this study indicate that increases in ammonia within the chamber, including short-term increases, can be quantitatively recovered.*

**Keywords.** *Ammonia volatilization, Dairy cattle, Livestock buildings.*

Accurate measurements of ammonia emissions from on-farm buildings and manure processing technologies such as composting are critical for establishing equitable regulations and for effective evaluation of mitigation techniques. However, quantifying these ammonia losses is a difficult task, particularly in relation to changes in air flow or ambient temperature. Ammonia losses are commonly estimated by measuring the concentration of ammonia in the air and multiplying that value times a representative air flow. Inaccuracies in ammonia measurements can result from differences in localized ammonia volatilization rates and incomplete mixing as well as inaccurate measurement technologies. Air flow is also difficult to measure accurately (Demmers et al., 2000; Phillips et al., 1998). In addition, air flow can directly influence ammonia volatilization. The two environmental factors that have the greatest impact on ammonia volatilization are air temperature and velocity (Anderson, 1995; Monteny et al., 1998).

Ammonia emissions from farms are a growing environmental concern (ApSimon et al., 1987; Bussink and Oenema, 1998; Phillips et al., 1999). Animal housing facilities and manure processing technologies are sources of these emissions (Bussink and Oenema, 1998; Phillips et al., 1999). However, the impact of air flow and ambient temperature on emissions from animal facilities has not been

studied under controlled conditions. Studies under controlled conditions have generally utilized scale-model facilities (Elzing and Monteny, 1997) or small chambers housing only manure (Anderson, 1995). Animals studies have generally exploited buildings with mechanical ventilation (Braam et al., 1997; Smits et al., 1995; Swierstra, 1995). However, following industry practice, air flow was generally varied in an attempt to maintain ambient temperature at a level comfortable to the animals. This interaction complicates attempts to isolate the impact of temperature and air flow on measured emissions. In addition, even mechanically ventilated houses often have large openings, which allow ammonia to dissipate to the atmosphere through diffusion and as a result of eddy currents. On windy days, the effect of eddy currents can be pronounced. Because of these and other problems, data from mechanically ventilated houses are generally noisy. These noisy data require that emissions be averaged over 24 h and that complex mathematical models be used to address data trends and to deal with changes in ambient temperature and air flow. Commonly, autoregressive time series models are used to fit log transforms of ammonia emissions data, and a linear correction factor, independent of air flow, is used to account for changes in ambient temperature (Monteny et al., 1998; Smits et al., 1995; Swierstra, 1995). However, potential interactions of temperature and emissions, and of temperature, air flow, and emissions were not addressed.

To allow ammonia emissions to be measured under conditions of precise and independent control of temperature and air flow, a large environmental chamber was constructed at Beltsville (Lefcourt et al., 2001). In this study, to validate the use of the chamber for ammonia studies, known quantities of ammonia were released in the chamber. The amount of ammonia recovered from each release was determined by multiplying the volumetric exhaust air flow by the increase in ammonia concentration in the exhaust air.

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## MATERIAL AND METHODS

Controlled quantities of ammonia were released in the environmental chamber from a small cylinder containing anhydrous ammonia. The actual quantity of ammonia released was determined by change in cylinder weight measured to an accuracy of 0.02 g. Ammonia concentrations in the exhaust air along with the volumetric air flow rate were used to determine amounts of ammonia recovered. Percentages of ammonia recovered were calculated by dividing amounts recovered by actual quantities released. The original experimental design was a  $3 \times 3 \times 3$  factorial with triplicate measurements; the factors were location of release, release rate, and air exchange rate.

### CHAMBER SPECIFICATIONS

The chamber, 7.4 m wide  $\times$  10.5 m long  $\times$  3.4 m high, was configured to house six dairy cows in tie stalls. Air temperature in the chamber did not deviate from the setpoint of 22.2°C by more than 0.1°C; relative humidity was maintained at less than 60%. Detailed descriptions of chamber design, operation, and control functions have been published (Lefcourt et al., 2001). Behind the stalls is a recessed gutter covered by grating which contains a scraping gutter cleaner. The cleaner was not activated during the time of the study. Air enters the chamber through six ducts in the ceiling above the gutter and exits through six ducts above the floor on the opposite wall (fig. 1).

### LOCATION OF AMMONIA RELEASE

Ammonia was released from one of three locations along the gutter (fig. 1). The release locations were directly under the ceiling supply ducts. However, for release position three air flow in the vicinity of the release position was affected by a large auxiliary air handler directly overhead; the air handler obstructed and dispersed the supply air flow before it reached the release cylinder.

### AMMONIA RELEASE MECHANISM

Anhydrous ammonia was transferred to a 150-mL sampling bottle (316L-HDF4-150; Swagelok, Ohio) as needed. Release was controlled using a stem valve (SS-14DKS4-S4-E; Swagelok) in series with an adjustable precision metering valve (SS-SS4-EPVH; Swagelok). A 35-cm section of polyvinyl chloride tubing (Tygon; 3.2 mm ID, 6.4 mm OD) was used to form a curly-q after the metering

valve to reduce effects of sputtering which often occurred when the stem valve was first opened. The entire assembly was supported in a vertical position using a plastic Lecture bottle holder (LB3581; Advanced Specialty Gas Equipment, N.J.). The instantaneous ammonia release rate is a function of the vapor pressure of ammonia in the sampling cylinder and the flow coefficient of the metering valve. Because the release of ammonia causes cooling of the sampling cylinder, the vapor pressure of the ammonia in the cylinder decreases over time resulting in a time-dependant decrease in the release rate. The three setpoints for the metering valve used in this study were approximately 0.04, 0.4, and 1.2 turns open.

### AMMONIA MEASUREMENT

Ammonia concentrations were measured using a Chillgard® RT Model 3800 Gas Monitor (Mine Safety Appliances Co., Cransberry Township, Pa.). The monitor specifications indicate a sensitivity of 1 ppm with an accuracy of  $\pm 1$  ppm. However, these specification are for instantaneous readings and the monitor output actual oscillates with a period of 1–2 min around the correct reading; thus, 10-min averages of monitor readings are more accurate than the specifications indicate. The monitor was housed in a drying oven at 37°C, and was calibrated using ammonia gas standards of 10.0, 20.9, and 49.5 ppm in nitrogen and ultra-pure nitrogen. To increase the resolution of the monitor, the low and high points used in the monitor set-up were exaggerated; i.e., the 0-ppm standard was input as 5 ppm and the 49.5-ppm standard was input as 90 ppm. The issue of the accuracy of the standards was addressed by requiring the vendor (BOC Gases, Murray Hill, N.J.) to certify the gas standards at weekly intervals and to ship the standards only after three sequential certifications were within 1 ppm. In addition, equal flows of the 0- and 20.9-ppm, and 0- and 10.0-ppm standards were combined to yield 10.5- and 5.0-ppm reference points. Reference points were not used for standard curve calculations. Calibrations were performed ever two weeks; on alternate weeks, the 10.0-ppm standard was run through the monitor. The monitor outputs a 0- to 10-V signal proportional to measured ammonia concentrations. A computer with a 12-bit analog-to-digital acquisition board was used to acquire readings at 2-s intervals and to calculate averages over 30-s periods (15 readings). The averages were recorded to disk. For calibrations, standard gases were monitored for at least 30 mins with data from the first 10 min discarded. Data averaged over 10-min intervals were used to generate a linear calibration equation by the method of least squares (fig. 2). The sampling line from the exhaust plenum was polyvinyl chloride lined with Teflon® (Tygon SE-200; 3.2 mm ID, 6.4 mm OD). Aluminum tape was used to bind self-regulating heating cable (SRF 5-1; Omega, Stamford, Conn.) to the sampling line and both were covered with foam pipe insulation. To allow for uniform sampling, air was sampled from two adjacent ports joined by an external “Y” connector. The ports were in the main exhaust plenum just after an elbow containing air deflectors (fig. 3). For graphs, ammonia measurements were low-pass filtered (Blackman, 64 points, 8-min time constant; Elliot, 1987).

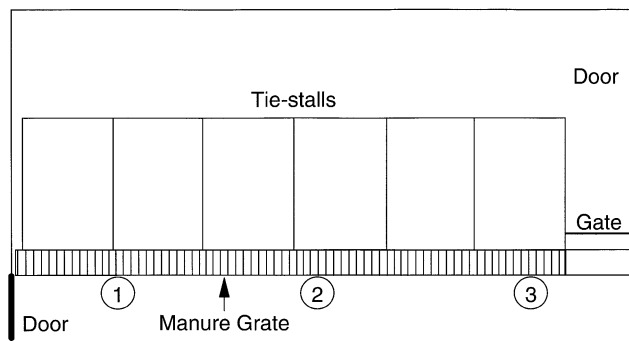
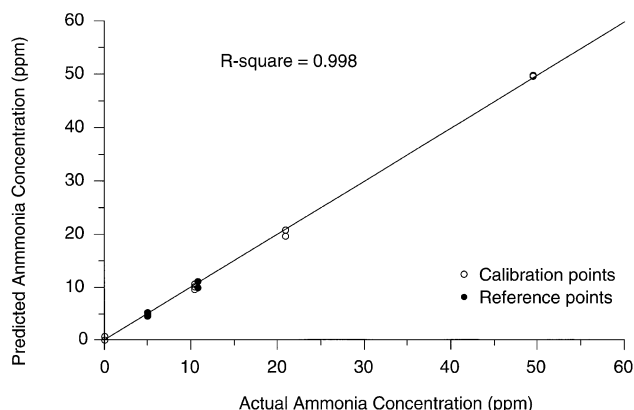


Figure 1. The three locations (numbered circles) in the environmental chamber from which ammonia was released.



**Figure 2.** Calibration curve for ammonia monitor. Averaged 10-min readings were used to generate a linear calibration equation by the method of least squares (open circles). Points generated by mixing calibration gases are shown for reference (closed circles).

### VOLUMETRIC AIR FLOW MEASUREMENT

Air flow was measured using a monitoring station located in the main exhaust duct and containing a pitot tube array in conjunction with two differential pressure transducers located adjacent to the station. The use of two transducers with different measurement ranges improves the accuracy of air flow estimates ( $\pm 1\%$ ; Lefcourt et al., 2001). Volumetric air flow setpoints were entered into the system controller, and the controller adjusted the setpoint so that the mass flow under the current operating conditions was equivalent to the mass flow of the entered volumetric air flow at 1 atm and 21.1°C (70°F). The three volumetric air flow setpoints used for this study were 42.4, 56.6, and 85.0 m<sup>3</sup>/min (1500, 2000, and 3000 CFM), which correspond to 10.5, 14.0, and 21.0 air exchanges per h.

### AMMONIA RECOVERY CALCULATIONS

The time sequence for individual trials was a 30-min baseline period, ammonia release for 30 min, a 30-min stabilization period, and a second 30-min baseline period (fig. 4). As determined to be appropriate by visual observation of results, the stabilization period was sometimes shortened by 10 min or extended by 15 min; trials with a low release rate and high air flow often returned to

baseline very quickly while trials with a high release rate and low air flow sometimes required additional time to return to baseline. The average increase in ammonia concentration due to a release was estimated by averaging measured concentrations over the combined release and stabilization periods and subtracting the average over the two baseline periods. The estimated ammonia release was calculated as: average concentration increase (ppm)  $\times$  duration of combined release and stabilization periods (normally 60 min)  $\times$  air flow (m<sup>3</sup>/min)  $\times$  0.7052 (ng/m<sup>3</sup>/ppm). The conversion factor 0.7052 ng/m<sup>3</sup>/ppm is the result of using the ideal gas law to determine the number of molecules in a cubic meter of gas at 1 atm and 21.1°C, dividing by 10<sup>6</sup>, and multiplying by the molecular weight of ammonia.

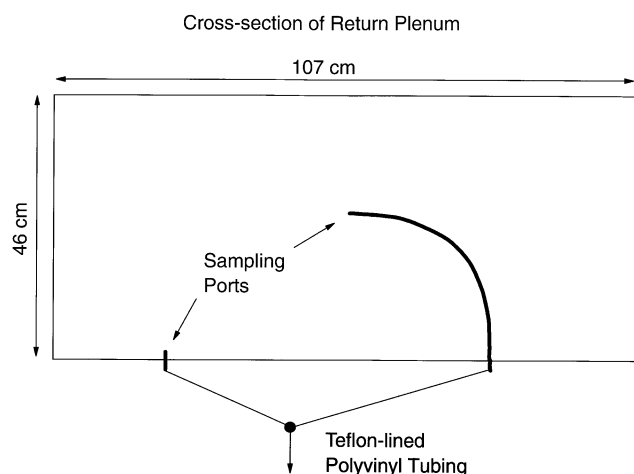
### STATISTICAL ANALYSES

Data were analyzed using PROC MIXED (SAS Institute, 1999) based on two strategies; first to determine whether air flow and release location affected recoveries and second to determine how best to adjust measured concentrations when the chamber is used to house animals. The first model included the factors FLOW (air flow rate), LOC (release location), and FLOW by LOC. Hypotheses were tested using Type II estimation functions. The initial experimental design also called for release rate to be a model factor; however, actual release rates varied too much to be considered as treatment levels. Hence, the initial model was modified to include the independent variables REL (release rate) and REL within LOC, as well as PPM (peak, digitally filtered, ammonia concentration during a release). The second strategy was to test practical models to be used to correct measured emissions when animals are housed in the chamber. The first practical model was identical to the initial model with the factors LOC and FLOW by LOC removed. The location factors were removed because with animals in the chamber, ammonia release would be distributed and not confined to strict locations. An alternate practical model was tested where air flow was incorporated in the model as a regression variable instead of as a treatment. The alternative model offers the advantage that predictions would be valid across the continuous range of air flows from 10.5 to 21.0 air exchanges per h.

## RESULTS AND DISCUSSION

Ammonia concentrations in livestock buildings generally range from 5 to 10 ppm, but can approach 70 ppm (Groot Koerkamp et al., 1998). Ammonia emissions rates from dairy barns range from 0.4 to 5 g per cow per hour (Braam et al., 1997; Swierstra, 1995). In preliminary trials with six heifers in the chamber, ammonia concentrations averaged around 10 ppm and ranged from 4 to 18 ppm over 24 h; emission rates were generally less than 6 g per cow per hour (personal observation). In this study, ammonia concentrations during release and recovery periods ranged from 1 to 27 ppm, well within the range commonly found in livestock buildings. Average release rates ranged from 0.1 to 0.7 g/min, which translates to 1 to 7 g per cow per hour.

Over 86 trials, the recoveries averaged  $105.1 \pm 0.8\%$ . The original experimental, a  $3 \times 3 \times 3$  factorial, called for 81 trials. However, it proved difficult to calibrate the ammonia release rate and it was decided that release rate should be an



**Figure 3.** Placement of air sampling ports in the return plenum.

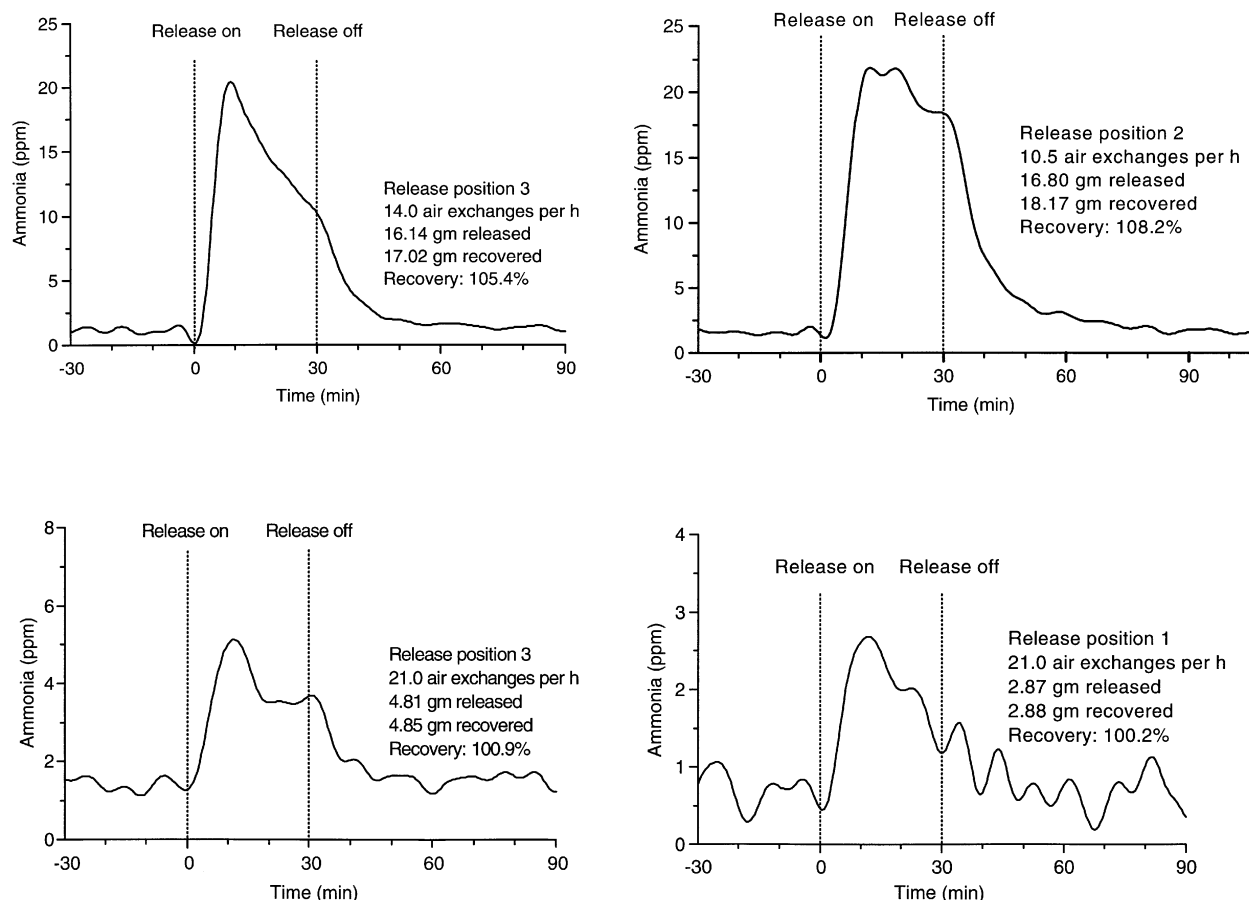


Figure 4. Examples of the time-course of measured ammonia concentrations in response to the release of ammonia in the environmental chamber.

independent variable instead of a treatment (see below). Problems with calibrating release rates resulted in five additional trials for a total of 86 trials.

The initial concept for the experimental design called for releasing ammonia at one of three different rates. A computer-based system was constructed to control the release of ammonia using a scale to continuously measure the weight of the ammonia release cylinder, a metering valve to limit maximum flow, and a solenoid to rapidly turn the release on and off to maintain the desired release rate. However, condensation due to the cooling effect of the ammonia release made weight measurements unreliable. As an alternative, it was decided that ammonia would be released using three different settings of the metering valve. The assumption was that the release rate would quickly rise to a peak and then slowly decline over time due to the cooling effect, and that the mass released over the 60-min release and stabilization periods would be reproducible. This assumption proved to be incorrect. Release rates varied presumably due to differences in the initial mass and temperature of the release cylinder and to play in the metering valve. The vapor pressure of ammonia decreases with decreasing temperature. As ammonia is released, the cooling effect of the release reduces the temperature of the cylinder. However, the decrease in temperature is also a function of the mass of the ammonia in the cylinder. When the cylinder is full, the increased mass mitigates the cooling effect of the ammonia release. In addition, when trials were conducted sequentially, the release system was sometimes below the ambient chamber temperature at the onset of release. A second factor

concerned low release rates. For low release rates, the precision metering valve was almost fully closed and the stem valve had to be used to adjust the release rate. More accurate control of the low release rates could have been accomplished by using a metering valve with two needle valves in series. At this point, the theoretical value of releasing ammonia at three fixed rates was reexamined. It was decided that a better experimental design would be to release ammonia using a spectrum of release rates, and to consider average release rate as an independent variable in the statistical model rather than as a treatment.

The two remaining factors in the experimental design were location of ammonia release and air flow. The three release positions along the gutter scrapper grating span the area where manure is most likely to accumulate when cows are housed in the chambers (fig. 1). The range of air flow was chosen to represent the normal range of conditions, which might be encountered in a barn, and to span a sufficient range to see if recoveries could be represented as a mathematical function of air flow. Examples of results from the 86 individual recovery trials are shown in figure 4. In general, there was a rapid rise in measured ammonia concentration a few minutes after the start of the release which reached a peak about 10 min into the release period. The peak was followed by a slow decline in concentration presumably associated with cooling of the ammonia release cylinder. When the release was halted, measured concentrations dropped rapidly and normally returned to baseline within 15 min. At the lowest air flow rate, 42.4 m<sup>3</sup>/min, the time to reach baseline was often extended

to 30 min (fig. 4). Concentration readings sometimes showed an oscillation with a period of 8–10 min (fig. 4). The amplitude of this oscillation occasionally approached 0.5 ppm; the amplitude, and even the existence, of this oscillation varied across trials conducted on the same day. The oscillations may be related to an intermittent problem with the air flow pump in the ammonia monitor. Another potential measurement artifact were transient drops in measured ammonia concentration around time zero (fig. 4). These drops occurred when the door to the chamber was opened. Presumably the resulting pressure pulse affected the stability of the monitor. The drops in readings were not due to an influx of building air as the presence of a manure scraping system in the building elevated ammonia concentrations above outside ambient levels.

Recoveries were found to depend both on release location and air flow (fig. 5). Recoveries from position three were lower than for positions one and two, and were higher at higher air flow rates. Statistical analysis of the data using the initial model, described above, showed that the interaction of FLOW by LOC was not significant and that PPM (peak ammonia concentration) was a better predictor of recovery than was REL (release amount). The interaction was removed from the model and, as the R-squared between PPM and REL exceeded 0.9 (table 1), REL was also removed from the model. For comparison, figure 5 shows recoveries as a function of REL and as a function of PPM at 14.0 air exchanges per h. Recoveries from position three were found to be significantly lower ( $P < 0.01$ ) than those for positions one and two (table 2). This discrepancy is most likely due to

presence of a large auxiliary air handler located above release position three. The air handler blocked the supply ducts above this release position and reduced air flow in the vicinity of release position three.

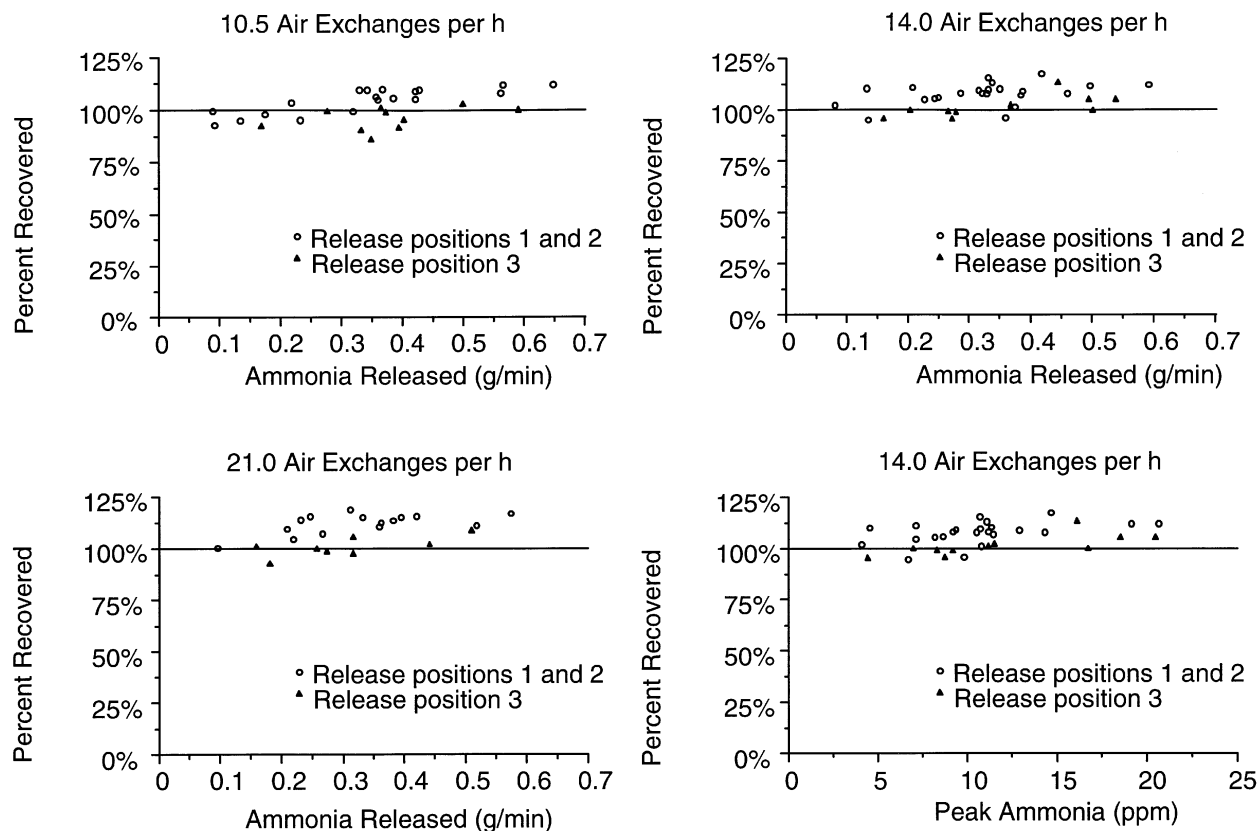
The increases in measured recoveries associated with increased air flow and increased amounts of ammonia released are probably due to more pronounced ammonia gradients across the chamber. The increased gradients in all likelihood result in proportionally higher concentrations of ammonia in the center of the plenum where the air velocity is highest; one of the two sampling ports is in the center of the exhaust plenum. The lower recoveries from position three support this hypothesis. Air flow to this release point is restricted by the presence of the auxiliary air handler, which allows for more uniform diffusion of ammonia in the chamber.

To explore the importance of location of release on recoveries, two additional models were tested; one with locations one and two combined as a single equivalent location and a second with location (LOC) removed from the

**Table 1. Correlations of peak, digitally filtered, ammonia concentrations (PPM) with quantities of ammonia released (REL)<sup>[a]</sup> by air exchange rate.**

Air Exchanges per h	R-Squared PPM by REL
10.5	0.93
14.0	0.93
21.5	0.92

<sup>[a]</sup> By FLOW, Model PPM = REL.



**Figure 5. Ammonia recoveries for individual trials as a function of the quantity of ammonia released by air flow (10.5, 14.0, or 21.0 air exchanges per h). For comparison, recoveries as a function of peak, digitally filtered, ammonia concentrations during the release period are shown for the case of 14.0 air exchanges per h.**

**Table 2. Percentage of ammonia recovered by release position.<sup>[a]</sup>**

Location	Percent Recovery
Position 1	109.0 ± 0.8
Position 2	107.8 ± 0.8
Position 3	99.5 ± 0.8

<sup>[a]</sup> Model recovery = flow loc PPM.

model (table 3). Comparison of results from the original and the two additional models indicate that a model based on FLOW level and maximum ammonia concentration during 30-min periods is a reasonable model to use to adjust measured ammonia concentrations when the chamber is used to house animals.

Instead of considering air flow as a treatment, it is possible to use flow as a regression variable. The statistical model where LOC was excluded as a factor was modified to substitute a polynomial expansion of flow; square-root of flow, actual flow, and flow squared as independent variables in place of the treatment FLOW. Flow squared was removed from the model due to lack of impact. The resulting estimated recoveries, PPM regression coefficient, and standard errors were identical to the predictions of the model that included FLOW as a treatment. Given that the predictions of the models are equally valid, the preferable model for practical use is the model where flow and the square-root of flow are treated as independent variables. This alternative offers the advantage that measured ammonia concentrations can be corrected over the entire range of air flows from 10.5 to 21.0 air exchanges per h.

#### CONSIDERATION OF BACKGROUND AMMONIA CONCENTRATIONS

One factor commonly not considered in estimations of ammonia emissions is background concentration of ammonia. Average ammonia concentrations over 24-h periods on days when the chamber was not used to conduct ammonia trials generally ranged from 0.6 to 1.2 ppm. Background readings were higher during the day; i.e., when recovery trials were conducted. During a period when corn in an adjacent field was cut for silage, the measured background concentration of ammonia approached 2 ppm. For the chamber, the average background of 0.9 ppm corresponds to 38.8 g over 24 h assuming 10.5 air exchanges per h. If this average background is ignored, daily emissions will be overestimated by 38.8 g. If instead, measurements are corrected for the average background of 0.9 ppm, but the background was really 0.6 or 1.2 ppm, the error over 24 h would be 12.9 g. However, in this case, the error would

**Table 3. Percentage of ammonia recovered by air exchange rate estimated using different linear models. Means are shown ±SEMs.**

	Model 1 (FLOW, LOC <sup>[a]</sup> , PPM)	Model 2 (FLOW, LOC, PPM)	Model 3 <sup>[b]</sup> (FLOW, PPM)
Air Exchanges per h			
10.5	98.0 ± 0.8	99.5 ± 0.8	99.6 ± 1.2
14.0	104.7 ± 0.7	106.2 ± 0.7	106.2 ± 1.0
21.5	109.1 ± 0.9	110.6 ± 0.9	110.3 ± 1.3
PPM regression estimate <sup>[c]</sup>	0.76 ± 0.09	0.76 ± 0.09	0.70 ± 0.13

<sup>[a]</sup> Positions 1 and 2 treated as a single equivalent location.

<sup>[b]</sup> Results were identical when the regression variables square-root of flow and actual flow were substituted for the treatment FLOW.

<sup>[c]</sup> Regression estimate of the effect of peak PPM on recoveries (mean peak PPM 11.56).

average to zero if numerous 24 h averages were themselves averaged. Thus, it is not necessary to continuously monitor baseline ammonia concentrations if the goal is to estimate average daily emission; however, it is still necessary to account for the average background in the estimation process. For short measurement periods, on the order of 30 to 60 min, the background concentrations become a more significant issue. The recovery calculations used in this study are very sensitive to background concentrations. At a release rate of 0.1 g/min, a 0.3-ppm error in the baseline results in an estimated recovery error of greater than 20%. At a release rate of 0.7 g/min, the error falls to about 3%. The recovery calculations reported in this study are based on measuring a baseline before and after the release of ammonia. In practice, it would be necessary to measure the baseline concentrations of ammonia in the air as it enters the chamber. Simultaneous measurement of ammonia concentrations in the intake and exhaust plenums would require the use of a second monitor as measurements from the monitor used in this study have to be averaged over at least 10 min to account for measurement oscillations. One alternative is to measure background (intake) ammonia concentrations periodically during the day at times when ammonia concentrations in the exhaust are known to be stable; e.g. during the night when animals are quiescent or during periods when animals are removed from the chamber for exercise. This procedure, particularly at the high emission rates commonly found on farms, should result in negligible errors due to baseline ammonia concentrations.

#### SUMMARY AND CONCLUSIONS

Due to physical limitations in the control of air flow and temperature, prior studies of ammonia emissions from cow barns have not been able to adequately address the effects of these environmental variables on emissions. Theoretically, both temperature and air flow are critical factors governing such emissions. The use of a large environmental chamber where temperature and air flow can be controlled and varied independently allows the effect of these environmental variables on emissions to be directly tested. The chamber can be used to test ammonia emissions from animals housed in the chamber and from manure processing technologies such as composting. The goal of this study was to demonstrate that ammonia released in the chamber could be recovered quantitatively, and to determine the influence of ammonia release rate, air exchange rate, and location of ammonia release on the accuracy of recoveries. The overall recovery of released ammonia for 86 trials averaged 105.1 ± 0.8%. Recovery estimates were improved by considering air flow and peak measured ammonia concentrations. Results from this study indicate that increases in ammonia within the chamber, including short-term increases, can be quantitatively recovered.

#### ACKNOWLEDGEMENTS

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